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IS 6605 (1972): Potassium Chromate [CHD 1: Inorganic Chemicals]



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Indian Standard
SPECIFICATION FOR
POTASSIUM CHROMATE

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 1

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SPECIFICATION FOR POTASSIUM CHROMATE

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Indian Standard

SPECIFICATION FOR POTASSIUM CHROMATE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 18 May 1972, after the draft finalized by the Inorganic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 Potassium chromate is chiefly used in the manufacture of pigments. It is also used in leather finishing, as a mordant in textile industry, in corrosion inhibition and as analytical reagent. It has a limited application in the manufacture of enamels.

0.3 Flame photometric method has been prescribed for the determination of sodium. Since widely different types of instruments are available for this determination, detailed instructions have not been given.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for potassium chromate.

2. GRADES

2.1 The material shall be of the following three grades:

- a) Technical (TECH),
- b) Pure, and
- c) Analytical Reagent (AR).

*Rules for rounding off numerical values (*revised*).

3. REQUIREMENTS

3.1 Description — Potassium chromates shall be in the form of lemon-yellow crystals with no water of crystallization. It shall be soluble in water.

3.2 The material when tested according to the methods prescribed in Appendix A shall comply with the requirements laid down in Table 1. Reference to the relevant clauses of Appendix A is given in col 5 of the table.

TABLE 1 REQUIREMENTS FOR POTASSIUM CHROMATE

SL No.	CHARACTERISTIC	REQUIREMENT FOR GRADE			METHODS OF TEST (REF TO CL No. IN APPENDIX A)
		TECH	PURE	AR	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Matter insoluble in water, percent by mass, <i>Max</i>	0.01	0.005	0.005	A-2
ii)	Moisture, percent by mass, <i>Max</i>	0.20	—	—	A-3
iii)	Potassium chromate (as K_2CrO_4), percent by mass, <i>Min</i>	98.5	99.50	99.50	A-4
iv)	Chlorides (as Cl), percent by mass, <i>Max</i>	0.10	0.005	0.001	A-5
v)	Sulphates (as SO_4), percent by mass, <i>Max</i>	0.30	0.05	0.01	A-6
vi)	Iron and aluminium oxides (as R_2O_3), percent by mass, <i>Max</i>	—	—	0.002	A-7
vii)	Calcium (as Ca), percent by mass, <i>Max</i>	—	0.005	0.005	A-8
viii)	Sodium (as Na), percent by mass, <i>Max</i>	—	—	0.025	A-9
ix)	pH of aqueous solution	—	8.6 to 9.0	8.6 to 9.0	A-10

4. PACKING AND MARKING

4.1 Packing — Technical grade of the material shall be packed in mild steel drums lined with polyethylene or gunny bags with polyethylene lining. Pure grade shall be packed in polyethylene bags which shall be placed in cardboard cartons. Analytical reagent grade shall be packed in bottles.

4.2 Marking — The containers shall be legibly and indelibly marked with the following information:

- a) Name and grade of the material;
- b) Net mass;
- c) Year of manufacture;
- d) Manufacturer's name and/or his recognized trade-mark, if any; and
- e) Lot number in code or otherwise to enable the batch of manufacture to be traced from records.

4.2.1 In case of AR grade of the material, complete chemical analysis in respect of the characteristics specified in Table 1 shall also appear on the label.

4.2.2 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. Presence of this mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard, under a well-defined system of inspection, testing and quality control during production. This system, which is devised and supervised by ISI and operated by the producer, has the further safeguard that the products as actually marketed are continuously checked by ISI for conformity to the standard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 The method of drawing representative samples of the material, number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Appendix B.

APPENDIX A

(Clause 3.2)

METHODS OF TEST FOR POTASSIUM CHROMATE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1960*) shall be used in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Specification for water, distilled quality (revised).

A-2. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-2.1 Procedure — Weigh accurately about 20 g of the material and transfer to a beaker. Dissolve in about 200 ml of water and filter through sintered glass crucible No. G4. Wash with water till the filtrate is colourless and collect the washings. Dry the crucible at $105 \pm 2^\circ\text{C}$ and weigh till constant mass is obtained.

A-2.1.1 Take the filtrate along with the washings in a 500-ml volumetric flask and make up the volume with water up to the mark. This shall be the *prepared solution* for analysis.

A-2.2 Calculation

$$\text{Matter insoluble in water, percent by mass} = \frac{M_1}{M} \times 100$$

where

M_1 = mass in g of the residue, and

M = mass in g of the sample taken for the test.

A-3. DETERMINATION OF MOISTURE

A-3.1 Procedure — Weigh accurately about 5 g of the material in silica or porcelain dish of about 75 ml capacity. Dry the contents in an oven at $125 \pm 5^\circ\text{C}$ to constant mass.

A-3.2 Calculation

$$\text{Moisture, percent by mass} = \frac{100 (M - M_1)}{M}$$

where

M = mass in g of the material taken for the test, and

M_1 = mass in g of the material after drying.

A-4. DETERMINATION OF POTASSIUM CHROMATE

A-4.1 Reagents

A-4.1.1 *Standard Potassium Permanganate Solution* — 0.05 N.

A-4.1.2 *Ferrous Ammonium Sulphate Solution* — approximately 0.1 N.

A-4.1.3 *Dilute Sulphuric Acid* — approximately 2 N.

A-4.2 Procedure — Pipette out 50 ml of the *prepared sample solution* (see A-2.1.1) in a 500-ml volumetric flask and make up the volume with

water. Take out 25 ml of the solution in a conical flask. Add 25 ml of dilute sulphuric acid and 25 ml of ferrous ammonium sulphate solution. Titrate the excess of ferrous ammonium sulphate against standard potassium permanganate solution. Conduct a blank titration also.

A-4.3 Calculation

$$\begin{array}{l} \text{Potassium chromate (as } K_2CrO_4 \text{),} \\ \text{percent by mass} \end{array} = \frac{(V_1 - V_2) \times N \times 129.46}{M}$$

where

V_1 = volume in ml of standard potassium permanganate solution used in the blank titration,

V_2 = volume in ml of standard potassium permanganate solution used in the titration with the material,

N = normality of standard potassium permanganate solution, and

M = mass in g of the material present in the aliquot.

A-5. DETERMINATION OF CHLORIDES

A-5.1 For Technical Grade

A-5.1.1 Reagents

A-5.1.1.1 Concentrated nitric acid — see IS : 264-1968*.

A-5.1.1.2 Silver nitrate solution — approximately 5 percent (m/v).

A-5.1.1.3 Dilute nitric acid — approximately 4 N.

A-5.1.2 Procedure — Take 200 ml of the *prepared sample solution*. Add 20 ml of concentrated nitric acid and heat to about 50°C. Add to this hot solution, sufficient volume of silver nitrate solution to precipitate chlorides completely. Protect the silver chloride precipitate from light by wrapping black paper around the container. Cool the solution and filter through a tared sintered glass crucible No. G4. Wash the precipitate first with dilute nitric acid to dissolve the adhering silver chromate and then with cold water. Dry the crucible and its contents to constant mass at $130 \pm 2^\circ\text{C}$.

A-5.1.3 Calculation

$$\text{Chlorides (as Cl), percent by mass} = \frac{24.7 \times M_1}{M}$$

where

M_1 = mass in g of silver chloride precipitate, and

M = mass in g of the material present in the aliquot.

*Specification for nitric acid (*first revision*).

A-5.2 For Pure and Analytical Reagent Grades

A-5.2.1 Apparatus

A-5.2.1.1 *Nessler cylinders* — 50 ml capacity.

A-5.2.2 Reagents

A-5.2.2.1 *Dilute nitric acid* — approximately 4 N.

A-5.2.2.2 *Silver nitrate solution* — approximately 5 percent (*m/v*).

A-5.2.2.3 *Standard chloride solution* — Dissolve 0.1649 g of ignited sodium chloride in 1000 ml of water. Dilute 100 ml of the solution to 1 litre. One millilitre of the diluted solution is equivalent to 0.01 mg of chloride (as Cl).

A-5.2.2.4 *Ammonium hydroxide* — 20 percent (*m/m*).

A-5.2.3 *Procedure* — Weigh accurately 0.20 g of the material, dissolve in 10 ml of water in a Nessler cylinder and add 1 ml of ammonium hydroxide and 1 ml of silver nitrate solution. Add 5 ml of dilute nitric acid and dilute to the mark. Carry out a control test in another Nessler cylinder using 1 ml of standard chloride solution for pure grade and 0.2 ml for AR grade of the material and the same quantities of other reagents. Compare the turbidity produced in the two cylinders.

A-5.2.3.1 The colour in the sample can be compensated for by superimposing a cylinder containing 0.2 g of the sample, 1 ml of ammonium hydroxide, 5 ml of dilute nitric acid, made up to the mark with water, over the cylinder containing the standard, and placing a cylinder containing 1 ml of ammonium hydroxide and 5 ml of dilute nitric acid behind the cylinder containing the sample. Both the turbidities are thus viewed through the same depth and colour of the solution.

A-5.2.4 The limit prescribed in Table 1 shall be taken as not having been exceeded if the opalescence produced with the material is not greater than that produced in the control test.

A-6. DETERMINATION OF SULPHATES

A-6.1 For Technical Grade

A-6.1.1 Reagents

A-6.1.1.1 *Concentrated hydrochloric acid* — see IS : 265-1962*.

A-6.1.1.2 *Rectified spirit* — see IS : 323-1959†.

A-6.1.1.3 *Glacial acetic acid* — see IS : 695-1967‡.

*Specification for hydrochloric acid (*revised*).

†Specification for rectified spirit (*revised*).

‡Specification for acetic acid (*first revision*).

A-6.1.1.4 Barium chloride solution — approximately 12 percent (m/v).

A-6.1.1.5 Dilute hydrochloric acid — approximately 1 percent (m/v).

A-6.1.2 Procedure

A-6.1.2.1 Concentrate 200 ml of the *prepared sample solution* to about 50 ml. Add 10 ml of concentrated hydrochloric acid, 15 ml of rectified spirit and 35 ml of glacial acetic acid. Boil for 15 minutes. Dilute to about 200 ml. To the hot solution add 20 ml of hot barium chloride solution in a slow stream with constant stirring, to precipitate sulphate. Continue boiling for 15 minutes and allow to stand for 4 hours. Filter the solution containing barium sulphate through a tared sintered glass crucible No. G4, Gooch crucible or a filter paper (Whatman No. 42 or equivalent). Wash with dilute hydrochloric acid till the filtrate is free from chromium salts and then with water till it is free from chlorides.

A-6.1.2.2 If the filtrate has been done through a sintered glass crucible, dry the precipitate at $110 \pm 5^\circ\text{C}$. If through Gooch crucible, dry the precipitate and ignite it over a burner or in the muffle furnace at 600 to 700°C for half an hour. In case of filter paper, after drying, ashing shall be done on a low flame and the precipitate then ignited over a burner or in a muffle furnace at 600 to 700°C for half an hour. Cool, add 2 drops of concentrated nitric acid and gently heat to drive off fumes. Ignite till constant mass is obtained.

A-6.1.3 Calculation

$$\text{Sulphates (as SO}_4\text{)}, \text{ percent by mass} = \frac{41.2 M_1}{M}$$

where

M_1 = mass in g of the ignited residue, and

M = mass in g of the material present in the aliquot.

A-6.2 For Pure and Analytical Reagent Grades

A-6.2.1 Reagents

A-6.2.1.1 Barium chloride solution — approximately 12 percent (m/v).

A-6.2.1.2 Concentrated hydrochloric acid — see IS : 265-1962*.

A-6.2.1.3 Rectified spirit — see IS : 323-1959†.

A-6.2.1.4 Sodium carbonate — solid.

A-6.2.1.5 Ammonium hydroxide — 20 percent (m/m).

*Specification for hydrochloric acid (*revised*).

†Specification for rectified spirit (*revised*).

A-6.2.2 Procedure

A-6.2.2.1 Take 250 ml of the *prepared sample solution*. Add 5 ml of concentrated hydrochloric acid. Heat to boiling and add 25 ml of barium chloride solution. Digest on a steam bath for 2 hours and let it stand overnight. If a precipitate is formed proceed as in **A-6.2.2.2**.

A-6.2.2.2 Filter, wash the precipitate with water till the washings are colourless and ignite. Fuse the residue in a platinum crucible with 1 g of sodium carbonate. Cool, extract with hot water and filter. Add 5 ml of concentrated hydrochloric acid to the filtrate, dilute to 75 ml, heat to boiling, add 10 ml of rectified spirit and heat on the steam bath till the solution is clear green or colourless. Dilute to 100 ml and add about 10 ml of glacial acetic acid. Heat to boiling, add 10 ml of hot barium chloride solution, digest on the steam bath for 2 hours and let it stand overnight. Filter, wash with hot water till the washings show no chlorides. Ignite, cool and weigh. Continue ignition till constant mass is obtained.

A-6.2.2.3 Carry out a blank test simultaneously.

A-6.2.3 Calculation

$$\text{Sulphates (as SO}_4\text{), percent by mass} = \frac{41.2 (M_1 - M_2)}{M}$$

where

M_1 = mass in g of the ignited residue obtained in test with the material,

M_2 = mass in g of the ignited residue obtained in the blank test, and

M = mass in g of the material present in the aliquot.

A-7. DETERMINATION OF IRON AND ALUMINIUM OXIDES (R₂O₃)**A-7.1 Reagents**

A-7.1.1 *Concentrated Hydrochloric Acid* — see IS : 265-1962*.

A-7.1.2 *Ammonium Hydroxide* — 20 percent (*m/m*).

A-7.1.3 *Ammonium Chloride* — solid.

A-7.2 Procedure — Take 25 ml of the *prepared sample solution*, add 2 ml of concentrated hydrochloric acid. Further add 4 g of ammonium chloride and 0.5 ml of ammonium hydroxide and boil. Filter, wash and ignite the precipitate. Cool and weigh till constant mass is obtained. Preserve the filtrate for determination of calcium in **A-8.2**.

*Specification for hydrochloric acid (*revised*).

A-7.3 Calculation

$$\text{R}_2\text{O}_3, \text{ percent by mass} = \frac{100 \times M_1}{M}$$

where

M_1 = mass in g of the ignited precipitate, and

M = mass in g of the material present in the aliquot of the solution taken.

A-8. DETERMINATION OF CALCIUM

A-8.1 Reagents

A-8.1.1 Ammonium Hydroxide — 20 percent (m/m).

A-8.1.2 Ammonium Oxalate Solution — 15 percent (m/v).

A-8.1.3 Concentrated Sulphuric Acid — see IS : 266-1961*.

A-8.1.4 Dilute Hydrochloric Acid — approximately 1 N.

A-8.2 Procedure — Acidify the filtrate preserved in **A-7.2** with dilute hydrochloric acid to about 4 pH. Boil and add 10 ml of ammonium oxalate solution. Bring the pH to 7 by adding ammonium hydroxide. Allow the precipitate to settle for 4 hours. Filter any precipitate present, wash it with water and ignite in a silica crucible. After cooling, add a few drops of concentrated sulphuric acid and reignite at 500°C. Cool and weigh till constant mass is obtained.

A-8.3 Calculation

$$\text{Calcium (as Ca)}, \text{ percent by mass} = 100 \times \frac{0.294 \times M_1}{M}$$

where

M_1 = mass in g of calcium sulphate precipitate, and

M = mass in g of the material taken for test in **A-7.2**.

A-9. DETERMINATION OF SODIUM

A-9.1 Qualitative Test — Dissolve 1 g of the material in 10 ml of water. Dip a clean platinum wire to the edge of a colourless Bunsen flame. When it is found to be colourless cool and dip it in the solution of the sample. Again introduce it into the edge of the colourless Bunsen flame. The presence of sodium radical is established if the characteristic yellow colour is imparted to the flame.

*Specification for sulphuric acid (*revised*).

A-9.2 If sodium is present, estimate it on a suitable flame photometer at 589 m μ according to the directions of the manufacturer of the apparatus (*see 0.2*).

A-10. DETERMINATION OF pH

A-10.1 Procedure — Dissolve 10 g of the material in 100 ml of carbon dioxide-free water. Determine pH of the solution by means of a suitable pH meter using glass and calomel electrodes.

A P P E N D I X B

(*Clause 5.1*)

SAMPLING OF POTASSIUM CHROMATE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling samples, the following precautions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the sample, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

B-1.5 The samples shall be placed in clean, dry and air-tight glass or other suitable containers, on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost but not completely filled with the sample.

B-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, the year of manufacture and other important particulars of the consignment.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material of the same grade from a single batch of manufacture shall constitute a lot. If

a consignment is declared or known to consist of different grades or different batches of manufacture, the containers belonging to the same grade and batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

B-2.2 The number of containers (n) to be chosen from a lot shall depend on the size of the lot (N) and shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

LOT SIZE	NUMBER OF CONTAINERS TO BE SELECTED
N	n
(1)	(2)
Up to 50	3
51 „ 200	4
201 „ 400	5
401 „ 650	6
651 and over	7

B-2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose random number tables* shall be used. In case such tables are not available, the following procedure may be adopted:

Starting from any container, count them in one order as 1, 2, 3,..... up to r and so on, where r is the integral part of N/n . Every r th container thus counted shall be taken out.

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples and Referee Sample

B-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected for sampling. The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given under 3.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions a small but equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 300 g.

This composite sample shall be divided into three equal parts, one for the purchaser, the second for the supplier and the third to be used as referee sample.

*See IS : 4905-1968 'Methods for random sampling'.

B-3.1.3 The remaining portions of the material from each container (after a small quantity needed for the formation of composite has been taken out) shall be divided into three equal parts, each part weighing not less than 10 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given in **B-1.7**. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be for the purchaser, the second for the supplier and the third shall be used as referee sample.

B-3.2 The referee sample, consisting of the composite sample (see **B-3.1.2**) and a set of individual sample (see **B-3.1**), shall be marked for this purpose and shall bear the seals of the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of potassium chromate shall be conducted on each of the individual samples constituting set of the test sample.

B-4.2 Tests for the remaining characteristics shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples — The mean and the range of the test results for the determination of potassium chromate shall be calculated as follows:

Mean (\bar{X}) = The sum of the test results divided by the number of test results, and

Range (R) = The difference between the maximum and the minimum values of the test results.

B-5.1.1 If ($\bar{X} - 0.6 R$) is equal to or greater than the minimum requirement of potassium chromate for the relevant grade, the lot shall be declared to have satisfied the requirement for potassium chromate for the relevant grade.

B-5.2 For Composite Samples — The test results on the composite sample shall meet the corresponding requirements specified in **3**.

B-5.3 A lot shall be declared as conforming to this specification if it satisfies the requirements for each of the characteristics listed in **3**.

B-5.4 If the requirements for any of the characteristics are not met, the lot shall be declared to have not satisfied the requirements of this specification.

INDIAN STANDARDS

ON

CHEMICAL REAGENTS AND GENERAL METHODS OF TESTS

IS:

2088-1962	Modified Gutzeit method of test for arsenic
2263-1962	Methods of preparation of indicator solutions for volumetric analysis
2316-1968	Methods of preparation of standard solutions for colorimetric and volumetric analysis (<i>first revision</i>)
2317-1963	Method for gravimetric determination of sulphates
2362-1963	Determination of water by the Karl Fischer method
3225-1965	Methods for preparation of buffer solutions
4016-1966	Density-composition tables for aqueous solutions of sodium hydroxide
4048-1966	Density-composition tables for aqueous solutions of sulphuric acid
4284-1967	Method for volumetric determination of iron
4285-1967	Method for volumetric determination of calcium
4542-1968	Colorimetric methods of determination of iron
4730-1968	Method for determination of density of liquids
5194-1969	Determination of nitrogen—Kjeldhal method
5298-1969	Determination of distillation range and distillation yield
5305-1969	Volumetric determination of phosphorus
5741-1970	Methods for determination of pH
5762-1970	Determination of melting point and melting range
5949-1970	Methods for volumetric determination of calcium and magnesium using EDTA
6361-1971	Methods of colorimetric determination of phosphorus

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